Applied Surface Science
Room: Hall D - Session AS-TuP

Aspects of Surface Analysis Poster Session

AS-TuP1 UNIFIT 2008 - Spectrum Processing, Analysis and Presentation Software for Photoelectron Spectra, ***R. Hesse***, *P. Streubel, R. Denecke*, University of Leipzig, Germany

The aim of the development of the program UNIFIT is to combine appropriate description of the spectra by adequate models, convenient data handling, and excellent numerical performance for fast calculation with versatile opportunities for data transfer, comfortable handling, extensive graphical design options and fast export of high resolution graphics. The advantage of the presented software is the complete treatment of the data from the measurement up to the presentation. (i) The three commonly applied models for fitting photoelectron spectra: product, sum, and convolution of Gaussian and Lorentzian functions will be discussed. In order to illustrate the differences of the models a Cu 3p spectrum was fitted with two independent single lines and an adjustable background consisting of a 3rd degree polynomial and a Shirley background. The theoretically expected intensity ratio of $3p_{3/2}/3p_{1/2}$ of 2:1 is well reproduced applying the sum or (correct) convolution model but not with the product function. (ii) The study of the band structure of solids demands the knowledge of valence-band edge and Fermi level position. We recommend improved methods for determination of these values. (iii) The reliability of the quantification from XPS data was improved using calibrated intensity scales of the photoelectron spectrometer ESCALAB 220 iXL. Two different sub-routines for estimating the transmission functions T(E) of different acquisition modes of any photoelectron spectrometers are integrated in the software UNIFIT 2008. (iv) The new software offers nine different presentations: measured and fitted spectrum, transmission function, Wagner plot, 3D-waterfall 0°, 3D-waterfall 45°, 3D-waterfall -45°, 3D-colour profile, 3D-presentation of fitted spectra and parameter plot. Extensive graphical design tools permit the individual creation of the presentations. The graphics may be exported as BMP or JPG images with a resolution of 600 dpi. The saved pictures may be easily inserted in each Power Point presentation or Word document. (v) The batch-processing submenu serves as a fast and comfortable treatment of parameter-dependent experimental series, e.g., depth profiles, angle resolved spectra etc. (vi) UNIFIT permits the calculation of fit parameter errors after peak fitting. According to the chosen option the user can calculate the errors with two different methods: matrix inversion or iterative calculation.

AS-TuP2 Test of the Consistency of Angle Resolved XPS Data for Depth Profile Reconstruction Using the Maximum Entropy Method, *A.J. Roberts, K Macak,* ***D.J. Surman***, Kratos Analytical Ltd, UK

Angle resolved XPS is a useful method for obtaining non-destructive quantification of thin (4-6nm) layers with good absolute depth resolution.[1] Although acquisition of ARXPS data with modern instrumentation is easy, determining the depth distribution of elements from the data is more challenging. The maximum entropy method (MEM) is a technique frequently used for solving the inversion problem in angle resolved XPS experiments. The MEMSYS algorithm used by Livesey and Smith[2] can provide an estimate of the optimum value of the regularising parameter (smoothness), of the error bars in the reconstructed profile and of the noise level in the experimental data set. While for the artificial ARXPS datasets the interpretation of these factors is straightforward, the fitting of the experimental data may be complicated by the sample structure which does not always agree with the assumptions postulated by the model used in MEM calculations : the electron transport in the sample must be determined by the inelastic scattering with the constant mean free path throughout the sample and the material density must be constant throughout the sample. The necessary condition for the consistency of experimentally measured data with the MEM model (successful fit) is that the Laplace transform of the compositional depth profile (LTCDP) calculated from the experimentally measured normalised intensity is a monotonically decreasing function for all measured elements. We have found an efficient algorithm which can estimate the LTCDP for elements with different inelastic mean free paths and thus provide an independent estimate of the noise scaling calculated by the MEMSYS algorithm and verify whether the MEM model is appropriate for the measured data. Here we present results from a number of samples and draw conclusions on the use of these methods to generate elemental and chemical-state depth profiles.

[1] P.J. Cumpson, J. Electron Specrosc. Rel. Phenom. 73 (1995) 25
[2] A. K. Livesey, G.C. Smith, J. of Elec. Spectroscopy 67 (1994) 439-961.

AS-TuP3 Analysis Area Determination in Small Area XPS, ***C.J. Blomfield***, *S.J. Hutton, S.C. Page, S.J. Coultas*, Kratos Analytical Ltd, UK

Currently, the accepted method of analysis area determination for small area XPS is to scan either the selected area spot or area illuminated by the X-ray beam over a well defined edge. The maximum signal is assumed to be 100% and the minimum signal 0%, the value used to determine the analysis area is then defined as the distance for the signal to change from 80% to 20% (or 84% to 16%) of these values.[1] This method has previously[2] been shown to produce total analysis areas approximately three times the diameter of that given by this “edge” measurement. An alternative approach of comparing the signal measured when an analysis is taken in the centre of an aperture of predetermined size has recently been shown to be another valid method for analysis area determination.[3] We have compared both of these methods for Kratos Axis Nova and Axis Ultra instruments which use a virtual probe approach, with the analysis area limited by both an area defining aperture and an angle defining iris to produce a Gaussian analysis area profile. The suitability of these methods and the implications to the true analysis area for the analyst will be discussed.

[1] ISO/TR 19319 Surface Chemical Analysis – Auger Electron Spectroscopy and X-ray Photoelectron Spectroscopy- Determination of lateral resolution, Analysis Area and Sample Area Viewed by the Analyser 2003
[2] Baer DR, Engelhard MH, Surf. Interface Anal 2000; 29 766
[3] Scheithauer U, Surf. Interface Anal 2008.

AS-TuP5 Characterization of X-ray Photocathode in Transmission Mode at 3keV, ***H. Ikeura-Sekiguchi,*** National Institute of Advanced Industrial Science and Technology (AIST), Japan*, T. Sekiguchi,* Japan Atomic Energy Agency (JAEA)*, M. Koike*, National Institute of Advanced Industrial Science and Technology (AIST), Japan

X-ray photoelectron emission microscope (X-PEEM) combined with synchrotron radiation is one of the most promising techniques for real-time surface spectromicroscopy. When X-PEEM is used as an x-ray detector for a transmission x-ray microscope, information on the subsurface properties of thin samples is obtainable with high spatial resolution. For the purpose of application to X-PEEM imaging in transmission mode, X-ray induced electron emission properties from photocathodes were investigated. Experiments were performed at beamline BL-2 of the AIST synchrotron radiation facility TERAS in Tsukuba. The BL-2 is equipped with an Si(111) double-crystal monochromator. Back-surface secondary electrons were detected using a microchannel plate (MCP, Hamamatsu Photonics K.K.). Photocathode thin layers of Al with higher quality on a kapton film were produced using the helicon plasma sputtering system. Thickness dependence of total electron (mostly secondary electron) emission yields from back-surface of Al film is measured at 3 keV photon energy. Secondary electron escape depth of Al was estimated based on semiempirical equations and suitable photocathode thickness of Al was experimentally evaluated. Capability of the imaging technique using the transmission photocathode will also be discussed.

AS-TuP6 Advanced Data Analysis for Surface Topography Characterization of Niobium Superconducting RF Accelerator Cavities, *H. Tian,* College of William & Mary*, G. Ribeill,* North Carolina State University*, C.E. Reece,* Thomas Jefferson National Accelerator Facility, ***M.J. Kelley***, College of William & Mary

An unprecedented number and scale of particle accelerator projects are getting underway, most notably the 30-km plus long International Linear Collider. The dominant technology is based on superconducting niobium radio-frequency (SRF) cavities, powered to ever-increasing acceleration gradients. The rf energy’s shallow penetration depth – few tens of nm – lends great importance to cavity interior surfaces. Interior surface etching to remove mechanical damage leaves surface topography, including protrusions of varying sharpness. These may promote RF magnetic field entry, locally quenching superconductivity, so as to degrade cavity performance. A more incisive analysis of surface topography than the widely-used average roughness is needed. We introduce a Power Spectral Density (PSD) approach based on Fourier analysis of surface topography data acquired by both stylus profilometry and atomic force microscopy (AFM). We report the evolution of the Nb surface PSD as a function of applied etching and polishing steps, resulting in a novel quantitative description of roughness. An important aspect of the impact of processing on SRF performance is made significantly clearer.

AS-TuP7 XPS Analysis of a Complex Metal Oxide Coatings on Stainless Steel: Depth Profiling, ***H.M. Meyer, III***, Oak Ridge National Laboratory

A low-temperature process was used for depositing complex metal oxide films onto stainless steel (SS304). Some films contained Fe, Cr, Si, and Ca, others contained Sm and Ce, and still others had Fe and Zr. The coating process consisted of dipping the SS coupons once or several times into a patented liquid followed by a thermal treatment at temperatures comparatively lower than those experienced during more conventional CVD or PVD coating processes. This liquid plus thermal treatment coating process produced surface films ranging from 50 to 500 nanometers (nm), depending on the number of dipping steps. Coated parts have been tested in a variety of environments ranging from corrosive to abrasive and have shown enhanced surface properties leading to better performance. Thermo Fisher Scientific's K-Alpha XPS instrument was used for characterizing the through-depth composition of these films. This instrument has a micro-focused mono-chromatic Al ka x-ray source (spot size 30-400 microns), Ar ion sputter gun, and a charge compensation system utilizing both low energy electrons and low energy Ar ions. Depth profiles were obtained on these relatively thick films (500 nm) at high acquisition rates by acquiring the data at low energy resolution (i.e. high pass energy). Post-profiling data processing allowed the low resolution data to be transformed to high energy resolution using the Avantage Data Processing software (v.3.85) and special energy de-convolution routines. This method allowed rapid data acquisition along with detailed determination of through-depth chemistry via high energy resolution core level data. The post-processed data will be compared to Auger depth profiles and XPS data obtained using high energy resolution data acquisition at much slower acquisition rates. Research sponsored by the Assistant Secretary for Energy Efficiency and Renewable Energy, Office of FreedomCAR and Vehicle Technologies, as part of the High Temperature Materials Laboratory User Program, Oak Ridge National Laboratory, managed by UT-Battelle, LLC, for the U.S. Department of Energy under contract number DE-AC05-00OR22725.

AS-TuP8 Structural Characterization of Ag Nanoparticles Embedded in TiO_2 Thin Films Prepared by Means of RF-Magnetron Sputtering, ***V. Moellmann***, University of Paderborn, Germany, *P. Keil, J. Zuo, H. Itani, T. Titz,* Max-Planck-Institut fuer Eisenforschung GmbH, Germany, *G. Grundmeier*, University of Paderborn, Germany

The combination of nanometer-sized metal particles and semiconductor matrix materials displays size and matrix dependend properties. These nanocomposite materials have been attracting much attention in recent years because of their unique catalytic behaviour, nonlinear optical properties or selective optical absorption and reflection. Usually, the synthesis route strongly influences the properties of the embedded nanoparticles, especially in terms of the size, structure, crystal morphology, degree of crystallinity and the nature of the sample surface, which are important for the functional properties of thin nanocomposite films. Silver nanoparticles embedded in TiO2-films have a high potential for applications in the fields of solar energy conversion, photocatalysis, chemical and biological sensing or antibacterial coatings. RF-sputtering method is one of the most feasible methods due to its inherent versatility and the capability of obtaining a homogeneous surface coverage at low temperatures under controlled processing conditions. In the present study, TiO2 films with embedded Ag nanoparticles were prepared by means of RF-magnetron sputter deposition and subsequent annealing. We applied grazing incidence X-ray absorption spectroscopy amongst other analytical methods including AFM or SKPFM to investigate the effects of deposition on the size and structure, crystal morphology and the degree of crystallinity of the embedded Ag nanoparticles as well as the TiO2 matrix material. The influence of the deposition parameters on the structure of the nanocomposite films and on its optical properties and applications like reversible photochromic transformations or antibacterial coatings are presented.

AS-TuP9 Deposition and Characterization of Aluminum Oxynitride Optical Coatings, ***Y. Liu***, *N. Gunda, R. Cooke, R. Raman*, Entegris Inc.

Aluminum oxynitride (AlON) has received extensive attention due to its composition-dependent tunability in mechanical and optical properties. Aluminum oxynitride has been identified as a potential candidate material to replace sapphire (single crystal aluminum oxide) in various optical applications. However, it is still relatively expensive to fabricate bulk AlON by the conventional ceramic processing technique. The cubic AlON phase in the bulk material is obtained by a high temperature-synthesizing process to retain the crystal structure, which gives it optical, IR transparency and other attractive properties. It would be of importance for a number of applications, if the benefits of AlON properties could also be achieved in thin films. This thin film could then be applied on a variety of base substrates cost effectively. In this work, aluminum oxynitride coatings were deposited by magnetron sputtering followed by thermal treatments at different temperatures to evaluate phase conversions. Microstructures and phase constitution of both as-deposited and thermally treated AlON coatings were characterized by scanning electron microscopy (SEM), atomic force microscopy (AFM) and grazing incidence X-ray diffraction (GID). X-ray photoelectron spectroscopy (XPS) was employed to study the AlON coatings with respect to the stoichiometric composition and atomic bonding states. Mechanical properties such as Young's modulus, hardness and coefficient of friction of AlON coatings were investigated by depth-sensing nano-indentation and nano-scratching experiments. Optical properties of AlON coatings were examined by measuring refractive index, reflectance and transmittance in both visible and infrared range. The mechanical and optical properties of AlON coatings were compared with the powder-processed AlON ceramics.

AS-TuP10 Quantitative Analysis of Si-Ge Alloy Films and Compositional Depth Profiling of Si/Ge Multi Layers Using SIMS, *K.J. Kim,* ***J.S. Jang***, Korea Research Institute of Standards and Science, *T.E. Hong,* Korea Basic Science Institute, *H.J. Kang*, Chungbuk National University, Korea

Although secondary ion mass spectrometry (SIMS) is a powerful technique for the in-depth analysis of solid materials, it is difficult to apply for the quantitative analysis of major components due to severe matrix effect. In addition, the different ionization probability caused a significance interface artefact in the interfaces between two metals due to different ionization yields.[1] However, if the variation of ionization probability is linear in a specific composition range, the interface artefact can be compensated.[2] A Si/Ge multilayer, Si-Ge alloy multilayer films and Si-Ge alloy films were grown on Si (100) wafers by ion beam sputter deposition. The compositions of Si-Ge alloy films were analyzed by SIMS and compared with the nominal compositions obtained by Rutherford backscattering spectroscopy. The quantification results were found to be highly improved by oxygen ions with higher energy. In SIMS depth profiles of Si/Ge multilayers using oxygen ion beam, the interfaces are difficult to be defined because of the interface artifacts in the Si/Ge and Ge/Si interfaces due to the matrix effect. However, the interfaces could be clearly defined after conversion of the profiles to composition profiles using relative sensitivity factor determined from a SIMS depth profile of a Si52.4Ge47.6 alloy film. The etching rate of a Si layer is much slower than that of Ge layer because of the formation of SiO2 layer during O2+ ion sputtering.

[1]K. J. Kim and D. W. Moon, Appl. Phy. Lett., 60, 1178-1180 (1992).

[2]K. J. Kim, D. Simons, and G. Gillen, Appl. Surf. Sci. 253, 6000-6005 (2007).

AS-TuP11 Quantitative Multi-Technique Analysis of Silicon Germanium Semiconductors, ***R.E. Davis***, *M. Hatzistergos, B.M. St. Lawrence, T.L. Tai, A.G. Domenicucci, A Madan, T.L. Pinto, P. Ronsheim, Z. Zhu, A.R. Turansky, J.R. Holt, M. Hopstaken, T.N. Adam*, IBM Corp.

In its continuing quest for higher density and performance, the semiconductor industry, in addition to shrinking circuit dimensions, has also turned to novel engineered materials. This paper will describe one such material, epitaxially grown SiGe, and the significant challenges it has brought both to process engineering as well as micro-analysis. It was noted that the mobility of carriers can be enhanced by the selective local application of stress, either compressive or tensile. One way compressive stress can be achieved for pFETs is by incorporating a controlled amount of Ge into the Si lattice, and has been the focus of much recent activity.[1-3] Precise control of this enhanced mobility requires quantitative monitoring of the SiGe, for composition, structure and stress. In addition to the normal process concerns about consistency, uniformity, and throughput, the SiGe deposition process exhibits behavior referred to as "loading effects". In essence, the composition, structure and deposition rate depend on the overall area of exposed Si, as well as its feature size and immediate vicinity. These requirements for analysis have necessitated the careful application of a variety of micro- and bulk characterization techniques, including AES, SIMS, AFM, RBS, EPMA, Ellipsometry, SEM, TEM, XRD, XRR, and Raman techniques. Results will be presented using the AES, XRD and SIMS techniques for this demanding application.

[1] "Design of High Performance PFETs with Strained Si Channel and Laser Anneal", Z. Luo, Y. Chong1, J. Kim3, N. Rovedo, B. Greene, S. Panda, T. Sato4, J. Holt, D. Chidambarrao, J. Li, R. Davis, A. Madan, A. Turansky, O. Gluschenkov, R. Lindsay2, A. Ajmera, J. Lee1, S. Mishra1, R. Amos, D. Schepis, H. Ng, and K. Rim, IEDM Tech Digest, 495 (2005).

[2] "Impact of In-situ C Doping on Implant Damage and Strain Relaxation in Epitaxial SiGe layers on Si", Jinping Liu,* Anthony Domenicucci,** Anita Madan,** Jinghong Li,** Judson Holt,**, Richard Murphy, Andrew Turansky, Robert E. Davis, Lindsay E. Burns, and John Sudijono*, MRS 2006 Spring Meeting

[3] "Systematic Characterization of Pseudomorphic (110) Intrinsic SiGe Epitaxial Films for Hybrid Orientation Technology with Embedded SiGe Source/Drain", Qiqing (Christine) Ouyang*, Anita Madan, Nancy Klymko, Jinghong Li, Richard Murphy, Horatio Wildman, Robert Davis, Conal Murray*, Judson Holt, Siddhartha Panda, Meikei Ieong and Chun-Yung Sung, MRS Proceedings 913, pp. 13-18 (2006).

AS-TuP12 Atmospheric Plasma Surface Modification Analysis by Energy Resolved Molecular Beam Mass Spectrometry and SIMS*, **P.J. Hatton**, Y. Aranda Gonzalvo, G.A. Cooke, T.D. Whitmore, D.L. Seymour, C.L. Greenwood, J.A. Rees*, Hiden Analytical, UK

Electrical plasmas can be readily produced at atmospheric pressure and have relatively low running costs. They are ideal for a variety of industrial process applications for many materials. Processing using non-thermal atmospheric plasma currently extends to areas such as surface cleaning and functionalisation, plasma activation, tissue engineering and sterilisation. To aid in understanding the mechanisms involved in plasma/surface interaction we present results of both plasma measurements and surface composition studies for a range of materials treated using an atmospheric dielectric surface barrier discharge (DSBD). The plasma properties were determined using an energy-resolved molecular beam mass spectrometer (ERMBMS). The surface compositions of the materials before and after treatment were compared using a static SIMS instrument. The dielectric surface barrier discharges were operated using helium gas and applied to molybdenum, silicon wafers and printed circuits. Silicon test pieces were treated using hydrofluoric acid to produce a strongly hydrophobic, hydrogen-terminated surface. The hydrophobicity of the surfaces could be significantly reduced by short exposure to certain plasma conditions. Static SIMS analyses of these surfaces showed a significant reduction in the observed SiH+ signal and an accompanying increase in the Si and the reactive silanol groups [Si-OH]. ERMBMS consists of an energy mass spectrometer (QMS) with a differentially pumped three-stage inlet system [1]. Mass spectra of the ions generated in the DBSD source show a high concentration of monoatomic and diatomic oxygen. Analysis of the ion energy reveals that the ions are fully thermalized, with energies close to 0.03 eV. ERMBMS analyses of the plasmas provided information on the relative contributions of ionic and radical species to the changes in the surface structure and the combination of data from the two diagnostic techniques contributes to our general understanding of such plasma/surface processing.

Reference [1] Y.Aranda Gonzalvo, T.D. Whitmore, J.A. Rees, D.L.Seymour, E. Stoffels, 2006 J.Vac.Sci. Technol., A 24 550. Keywords: energy mass spectrometer, dielectric surface barrier discharge, atmospheric plasmas, SIMS.

AS-TuP13 A Mutual Calibration Method to Certify the Thickness of Nanometer Oxide Films*, **K.J. Kim**, Y.S. Kim, J.S. Jang, J.W. Kim*, Korea Research Institute of Standards and Science

The reliable measurement of ultra-thin gate oxide thickness below 1 nm is one of the most important analysis issues for the next generation semiconductor devices as shown in the recent international technology roadmap (ITRS). X-ray photoelectron spectroscopy (XPS) is an ideal candidate for the thickness measurement of ultra-thin films because of the surface sensitivity due to the shallow detection depth of low energy photoelectrons. However, XPS needs a standard film to determine the electron attenuation length because XPS is not an absolute method to determine the original thickness of overlayer films. In a recent study on the measurement of SiO2 film thickness on a silicon substrate, the thicknesses measured by various methods showed large offset values, giving an apparent thickness when the real thickness is extrapolated to zero. Compensation of these offset values is a key solution for the establishment of the traceability in the measurement of SiO2 film thickness. In this study, a mutual calibration method is suggested as a new method to certify the thickness of SiO2 films on Si by compensating for the offset values. In a linear plot of the thicknesses measured by x-ray photoelectron spectroscopy (XPS) and transmission electron microscopy (TEM) of a series of SiO2 films with different thicknesses, the offset value of TEM and the thickness scale of XPS can be mutually calibrated. Using this method, the XPS photoelectron attenuation length can be well defined and XPS becomes traceable in the measurement of the thickness of SiO2 films.

AS-TuP14 Imaging Surface Organics via Single Photon Secondary Neutral Mass Spectrometry with Tunable Synchrotron VUV*, **L.K. Takahashi**, J. Zhou, M. Ahmed,* Lawrence Berkeley National Laboratory*, S.R. Leone,* University of California, Berkeley*, K.R. Wilson*, Lawrence Berkeley National Laboratory

Elemental chemical imaging of surfaces on the sub-micron scale with Secondary Ion Mass Spectrometry (SIMS) has yielded much new and tantalizing information. However, the method is limited in its application to more fragile organic molecules due to extensive fragmentation, which results in complicated mass spectra that are difficult to analyze. Furthermore, the sensitivities of different chemical species in SIMS are highly dependent on the local chemical environment, meaning that relative intensities in the mass spectra do not reflect the actual chemical abundances at the surface. In Secondary Neutral Mass Spectrometry (SNMS), ejected neutrals are post-ionized, effectively decoupling the desorption and ionization steps. The resulting mass spectra are less dependent on the local chemical environment. Fragmentation of organic molecules, however, has remained a problem. The ionization potentials of most organic molecules are around 9-11 eV, and commercial tabletop lasers usually have lower photon energies than this, necessitating multiphoton ionization. This ionization method imparts excessive energy to the neutral molecules which can fragment organic chemical species. Here we present a new method utilizing continuously tunable VUV synchrotron light for single photon post-ionization of secondary organic neutrals. By ionizing just above the threshold, it has been shown that fragment free mass spectra of organic molecules can be obtained. The tunability of the light also allows for isomer identification by their characteristic ionization energies and associated fragments. While the experiment is still in its early stages, preliminary results have yielded unique photoionization efficiency curves for different organic molecules. With further improvements, single photon VUV-SNMS can become a powerful tool capable of providing localized chemical information on real-world heterogeneous organic systems such as microbial cells and aerosols.

AS-TuP16 Effect of Polymer MW on XPS Valence Band Spectra*, **J.L. Fenton**, J. Chen*, The Dow Chemical Company

The molecular weight of a polymer can alter the properties of films as low molecular weight components migrate to the polymer surface. XPS (x-ray photoelectron spectroscopy) is an ideal method to characterize the surface for the presence of low molecular weight polymers. XPS valance band spectra have been shown to be an excellent method to quantify the amount of propylene or ethylene at the surface of ethylene-propylene blends. Valance band spectra can also be used to detect changes due to secondary structure. The work presented here shows how XPS can identify the changes in the valance band due to MW.

AS-TuP17 Temperature Effect on Chemical and Physical Stability of Low Pressure Plasma Polymerised Coatings for Biological Applications, ***S.I. Cho**,* Pusan National University, South Korea*, M. Dhayal*, University of Washington

The effects of substrates heating on chemical and physical stability of plasma polymerised acrylic acid films were studied. The change in surface chemistry and surface morphology were characterised by X-ray photoelectron spectroscopy (XPS) and atomic force microscopy (AFM). XPS result showed that C(=O)OX functionality was unstable with the substrate heating temperature whereas C-OX and C=O functionalities were quite stable for 400oC. Small changes in the surface roughness were observed when substrate was heated upto 100oC. Higher temperature heating had created well structured patterns at the surface. Effect of these patterns and change in surface functionalities on cell growth and no-specific adsorption of proteins will be discussed.

AS-TuP18 The Electronic Structure of Pristine Copper-Hexadecafluorophthalocyanine (F16CuPc) from Resonant X-ray Emission Spectroscopy*, A. DeMasi, L.F.J. Piper,* Boston University*, **Y. Zhang**,* University of Nevada, Las Vegas*, K.E. Smith*, Boston University

Metal-based phthalocyanines (M-Pc) have become both fundamentally and technologically important.[S. Heutz et al., Adv. Mat. 19, 3618 (2007)] Here we report of recent synchrotron-based soft x-ray spectroscopy measurements of copper hexadecafluoro phthalocyanine (F16CuPc). Resonant x-ray emission spectroscopy (RXES) enables bulk, element and orbital-specific information of states near the Fermi level. We have examined the C, N, and F K-edges of thin films deposited in-vacuo, measured within our experimental endstation at the undulator beamline X1B at the National Synchrotron Light Source. Direct comparisons are made to earlier x-ray absorption and emission spectroscopy measurements of CuPc.[J. Downes et al., Chem. Phys. Lett. 390, 203 (2004)]. We focus on the C K-edge RXES of F16CuPc, and make comparisons with recent ultraviolet photoemission spectra and spin-polarized theoretical computations of non-fluorinated CuPc and F16CuPc.

AS-TuP19 XPS Analysis of Organic Materials Etched by Charged Water Droplet Impact*, **Y. Sakai**,* University of Yamanashi, Japan*, Y. Iijima,* JEOL Ltd.*, R. Takaishi, D. Asakawa, K. Hiraoka*, University of Yamanashi, Japan

A giant cluster ion impact method, electrospray droplet impact (EDI) ionization, has been developed for matrix-free secondary ion mass spectrometry.[1] EDI utilizes the electrospray charged water droplet as the extremely high-momentum projectiles. The charged water droplets formed by electrospraying 1M acetic acid aqueous solution at atmospheric pressure are passed through an orifice into the vacuum chamber, transported into a quadrupole ion guide, accelerated by 10kV after exiting the ion guide and impact on a target. Roughly, the charged water droplets may be represented as $[(H_2O)_{90000}+100H]^{100+}$ with mass of~1.6×10^6 u. The kinetic energy of impacting projectile is ~10^6 eV.[2] The polymer samples (PET, PMMA, PS) etched by EDI were evaluated by X-Ray Photoelectron Spectrometer (XPS: JEOL JPS-9200). A comparative study for PET, PMMA and PS etched by

Ar^+ ion and EDI was made. For the surfaces of PET, PMMA and PS etched by Ar^+ ion, the deoxygenation and graphitization were found. The degradations of the polymers by the ion sputtering are slightly suppressed by using the low energy ion or the cluster ion. But, the effect of suppressing degradations was depended on the polymers. On the other hand, the deoxygenation, and graphitization were not observed on the surfaces of PET, PMMA and PS etched by EDI. It supposed that the kinetic energy of impinging water droplet at the moment of impact on the target by EDI is efficiently converted to the internal energies of water molecules in the selvedge of the colliding interface. The rest is dissipated as shock waves propagating through the target and the water droplet. This verifies that EDI is capable of no degradations of the polymer surface etching. The other experimental results of EDI applied to metals, semiconductors will be given. Acknowledgement: This work was supported by the Japan Science and Technology Agency.

[1]K.Hiraoka, D.Asakawa, S.Fujimaki, A.Takamizawa, K.Mori, Eur.Phys. J. D 38 (2006) 225
[2]K.Hiraoka, K.Mori, D.Asakawa, J.Mass Spectrom., 41 (2006) 894.

AS-TuP20 XPS Study of Nitrogen Chemical Structure in DNA and Related Molecules*, **I. Ishizaki**, N. Sanada, S. Iida, M. Suzuki, Y. Ohashi,* ULVAC-PHI, Inc., Japan*, G. Hayashi, K. Nakatani*, Osaka University, Japan

Although recent XPS instrumental developments would provide high S/N ratio and much improved resolution for better interpretation of chemical structures,[1] relatively little application has been reported for bio materials including DNA. It has been reported that XPS N 1s lines of DNA nucleobases were affected by their chemisorption on Cu, and the peak shift was assigned as the dehydrogenation.[2] May et al.[3] reported in detail on XPS lines for several kinds of DNA and related materials, however, assignment of the N 1s lines in DNA was not fully elucidated as far as our knowledge. In this study, therefore, we studied nitrogen chemical structure of DNA-related molecules using XPS, cluster TOF-SIMS, and DFT calculations. The DNA-related molecules reported here include adenine (A), guanine (G), thymine (T), cytosine (C), and hypoxanthine (I), and their nucleotides, poly(nucleotides), single and double strained DNA molecules, and a DNA microarray. XPS N 1s lines had little differences between nucleobase and nucleotide for each series. On the other hand, they were sharply different between nucleotides and poly(nucleotides) for A, G, and C, but no obvious differences are found for T and I. Comparative study with G and I bases indicated that primary amine ($-NH_2$) group in the molecule played an important role on the chemical state changes of nitrogen. DFT calculation results of model nucleobase crystals and single-strained DNAs will also be discussed.

[1] for example, M. C. Biesinger, et al. Surf. Interface Anal., 36 (2004) 1550.
[2] M. Furukawa et al. Surf. Sci., 601 (2007) 5433.
[3] C.J. May, et al. Anal. Chem., 76 (2004) 1114.

AS-TuP21 Analysis of DLC Fine Structure on Sliders by Using Second Derivative Carbon KLL Auger Spectra*, **K.W. Wierman**, F.Y. Chen*, Seagate Technology

Present hard disc drive (HDD) manufacturing technology uses diamond-like carbon films (DLC) as overcoats for both head and media. These DLC films function to protect underlying magnetic metals from wear and corrosion both during the manufacturing process and throughout the design lifetime of the drive. Recording head DLC film thickness can vary from ~15 to ~30 Å while typical values of DLC films for magnetic media are in excess of 30 Å. Auger analysis is particularly suitable for overcoat studies due to the surface sensitivity of the technique. In this study an Omicron high resolution Auger/SEM system was utilized to investigate the DLC at the head air bearing surface. Carbon's Auger spectrum is particularly sensitive to the type of bond hybridization.[1] Graphite, diamond, and silicon carbide have distinctive Auger CKLL spectra and its fine structure that can be used to identify the form of carbon at the surface. A second derivative of the Auger spectrum was utilized to remove the background and highlight the carbon fine structure peak locations (maximum negative curvature).[2] To remove the surface hydrocarbons a 15 second 0.3keV Ar etch was utilized. W. Zhu et. al. showed that by reducing the Ar beam energy to 0.3keV and limiting the total flux to 1.0 x 10^{14} ions/cm^2 an effective cleaning of hydrocarbon contaminates from the surface of CVD diamond films was accomplished without negatively impacting the diamond carbon bond structures.[3] In-situ XPS data on DLC grown films on coupons was also completed for comparison of the carbon bond structure.

[1]T.W. Haas, J.T. Grant, and G.J. Dooley III, J. Appl. Phys., 43, (1972) 1853.
[2]Lascovich and A. Santoni, App. Surf. Sci., 103, (1998) 245.
[3]W.Zhu, J.E. deVries, M.A. Tamor, K.Y. Simon Ng, Surface Coatings Technology, 71, (1995) 37.

AS-TuP22 Root-Cause Failure Analysis of Anneal-Chamber Reflector Plates using Advanced XPS and TEM/EDX Applications*, **C. Lazik**, M. Jin, Y. Uritsky, L. Terry*, Applied Materials, Inc.

Delamination failures were observed on reflector plates in Vantage RTP systems during start-up in late-2006. Root-cause analysis was required to complete the current tool sign-off and to identify suspect parts/spares in the field. The reflector plate supplier revealed they had upgraded their e-beam evaporation chamber in June, 2006, but that the coating characteristics, reflectivity and adhesion strength, had not changed. The product support team provided reflector plates manufactured before and after the e-beam evaporation chamber upgrade to the Defect and Thin-Film Characterization Laboratory (DTCL) to identify any differences in the coatings that may explain the failure. Using X-ray Photoelectron Spectroscopy (XPS) and Transmission Electron Spectroscopy (TEM), DTCL identified titanium poisoning within a ~30A oxide layer at the base of the adhesion film on all of the failed parts. Results enabled the product support team to pinpoint the failure mechanism, locate the contamination source in the supplier's hardware, and implement new quality control inspection tests/procedures for new, incoming reflector plates.

AS-TuP23 Microstructural Transformations of Ultrathin TiDy/Pd Bi-Layer Films Evaporated on Si(100) and Quartz Induced by Vacuum Annealing*, **E.G. Keim***, University of Twente, The Netherlands*, W. Lisowski,* Polish Academy of Sciences*, M.A. Smithers*, University of Twente, The Netherlands

Thin TiD_y/Pd bilayer films[1] can be applied as a useful source of deuterium used in chemical and energetic reactions. Evolution of hydrogen from such material is realized by high-temperature annealing. Main question is to what degree structural changes occur within both the substrate and the ultrathin TiD_y/Pd bi-layer film as result of annealing induced decomposition of the titanium deuteride phase. In this paper we present the results of a combined SEM/TEM study dealing with these questions. The results are compared for two ultrathin TiD_y/Pd films evaporated on quartz and Si(100), respectively. Ultrathin TiD_y/Pd films (10 - 20 nm thick TiDy films covered by 10 - 20 nm thick Pd films) were prepared in a UHV glass system.[2] Decomposition of the titanium deuteride phase in the TiDy/Pd film during annealing, was monitored in situ by TDMS,[3] all other analysis ex situ. It was found that annealing of the TiD_y/Pd films, evaporated on various substrates, leads to significant transformations within the films studied. SEM images disclosed various stages of rearrangement of the surface morphology in both films. Energy Filtered TEM analyses of cross-section images as well as EDX point analyses revealed extensive inter-diffusion of Ti and Pd within both Ti-Pd bi-layer films. Moreover, the EF-TEM Si elemental map displayed also a significant penetration of Si atoms from the Si substrate into the Ti area of the Ti-Pd film.

[1] W. Lisowski, E.G. Keim, A.H.J. van den Berg, M.A. Smithers, Anal. Bioanal. Chem. 385 (2006) 700.
[2] W. Lisowski, Vacuum, 54, 13 (1999).
[3] W. Lisowski, E.G. Keim and M.A. Smithers, J. Vac. Sci. Technol. A21, 545 (2003).

AS-TuP24 Analysis of the Line Shape on Layer-Resolved Photoemission Signals in Determining the Absolute Coverage of Atomically Flat Metallic Thin Films*, **D.-A. Luh**,* National Central University, Taiwan*, C.-M. Cheng, K.-D. Tsuei*, National Synchrotron Radiation Research Center, Taiwan

An important application of nanotechnology is the construction of nanodevices for specific problems. Size is typically the parameter that engineers vary while adjusting the physical properties of nanodevices. Because of electronic confinement from the boundary, the properties of a nanostructure depend strongly on its size; this effect is called the quantum size effect (QSE), which has been reported on various physical properties. To study the QSE properly, one must determine and control the size of nanostructures with an atomic resolution. Failure to do so not only makes the analysis of the QSE less confident, but also makes the process of manufacturing nanostructures unreliable. To address this issue, we report a technique to determine film coverage precisely by analyzing layer-resolved signals in one photoemission spectrum. We demonstrate that the coverage of a metallic thin film on the (111) surface of noble metals is determined precisely on analyzing its layer-resolved confined states measured with angle-resolved photoelectron spectra. In our work, the surface state on a Ag film of 1~4 ML and the quantum well states on a Ag film of 7~13 ML on Au(111) were analyzed with atomic resolution. Through analysis of the line shape with tabulated binding energies, we determined precisely the absolute Ag coverage of a Ag/Au(111) film on analyzing a single spectrum. For this technique to work, photoemission cross sections for signals corresponding to adjacent thicknesses are assumed to be similar, and the intensities of the signals are directly proportional to the domain area of their corresponding film thicknesses. This assumption disagrees with previous authors who reported the variation of photoemission cross sections with photon energy in many systems. To investigate the prospective inconsistency, we

performed energy-dependent photoemission measurements on atomically flat thin films with well-controlled coverage. Our results show that the line-shape analysis is valid with an absolute error in the measured absolute coverage within 0.1 ML for a Ag film on Au(111) when the photon energy is appropriately chosen. The experimental procedure employed in our work not only validates the line-shape analysis but also serves as a routine to determine an appropriate photon energy for the line-shape analysis. The line-shape analysis with absolute error possesses great advantages over other techniques with relative error, especially for a higher film coverage.

AS-TuP25 3-Dimensional XPS Imaging of Surface Nano-structures; A New Technique*, **S. Tougaard**, S. Hajati*, University of Southern Denmark

XPS energy spectra vary characteristically with the depth distribution of electron emitting atoms on the nano-scale. This is the basis for the by now well known and widely used method[1] to non-destructively determine atomic depth distributions with nano-meter resolution by analysis of the inelastically scattered electrons associated with the XPS peak. A new algorithm which is suitable for automation was suggested recently.[2] For each XPS signal, this algorithm determines the total amount of the corresponding atoms within the outermost ~ 10 nm and it also determines their depth distribution. The validity of the algorithm was demonstrated experimentally by comparison to more elaborate quantification methods.[3] In addition, software that can automatically analyze several thousand spectra corresponding to the situation in XPS imaging is developed. The software produces nondestructively a 3-D image of the surface with nanometer depth resolution. The practical applicability for XPS imaging was recently demonstrated.[4-5] As an example we have demonstrated a quantitative test[4] of the algorithms ability to produce images of Ag taken from a series of samples with increasing thicknesses of plasma patterned Octadiene (2, 4, 6 and 8nm) on Ag substrates. The obtained images of the amount of silver atoms in the outermost few nano-meters of the samples were in good agreement with the nominal thicknesses. For a given sample, different sectioning of depth distributions of atoms were made which clearly prove the ability of the method for quantitative and nondestructive 3-D characterization of nano-structures. In ref [5], 3D images of thermally patterned oxidized silicon made through a photolithographic mask were produced and it was shown that 3-D images of the Si, O, and C atoms were complementary. In the talk we will summarize the technique and discuss its limitations and capabilities.

[1]S. Tougaard Surf. Interf. Anal. 26, 249 (1998)
[2]S. Tougaard, J. Vac. Sci. Technol. A21, 1081 (2003)
[3]S. Tougaard, J. Vac. Sci. Technol. A23, 741 (2005)
[4]S. Hajati, S. Coultas, C Blomfield and S. Tougaard, Surf. Interf. Anal. 40, 688 (2008)
[5]S. Hajati, J. Walton, N. Fairley, and S. Tougaard, Surf. Sci. (2008). In press.

Authors Index

Bold page numbers indicate the presenter

— A —

Adam, T.N.: AS-TuP11, 2
Ahmed, M.: AS-TuP14, 3
Aranda Gonzalvo, Y.: AS-TuP12, 3
Asakawa, D.: AS-TuP19, 3

— B —

Blomfield, C.J.: AS-TuP3, **1**

— C —

Chen, F.Y.: AS-TuP21, 4
Chen, J.: AS-TuP16, 3
Cheng, C.-M.: AS-TuP24, 4
Cho, S.I.: AS-TuP17, **3**
Cooke, G.A.: AS-TuP12, 3
Cooke, R.: AS-TuP9, 2
Coultas, S.J.: AS-TuP3, 1

— D —

Davis, R.E.: AS-TuP11, **2**
DeMasi, A.: AS-TuP18, 3
Denecke, R.: AS-TuP1, 1
Dhayal, M.: AS-TuP17, 3
Domenicucci, A.G.: AS-TuP11, 2

— F —

Fenton, J.L.: AS-TuP16, **3**

— G —

Greenwood, C.L.: AS-TuP12, 3
Grundmeier, G.: AS-TuP8, 2
Gunda, N.: AS-TuP9, 2

— H —

Hajati, S.: AS-TuP25, 5
Hatton, P.J.: AS-TuP12, **3**
Hatzistergos, M.: AS-TuP11, 2
Hayashi, G.: AS-TuP20, 4
Hesse, R.: AS-TuP1, **1**
Hiraoka, K.: AS-TuP19, 3
Holt, J.R.: AS-TuP11, 2
Hong, T.E.: AS-TuP10, 2
Hopstaken, M.: AS-TuP11, 2
Hutton, S.J.: AS-TuP3, 1

— I —

Iida, S.: AS-TuP20, 4
Iijima, Y.: AS-TuP19, 3
Ikeura-Sekiguchi, H.: AS-TuP5, **1**
Ishizaki, I.: AS-TuP20, **4**
Itani, H.: AS-TuP8, 2

— J —

Jang, J.S.: AS-TuP10, **2**; AS-TuP13, 3
Jin, M.: AS-TuP22, 4

— K —

Kang, H.J.: AS-TuP10, 2
Keil, P.: AS-TuP8, 2
Keim, E.G.: AS-TuP23, **4**
Kelley, M.J.: AS-TuP6, **1**
Kim, J.W.: AS-TuP13, 3
Kim, K.J.: AS-TuP10, 2; AS-TuP13, **3**
Kim, Y.S.: AS-TuP13, 3
Koike, M.: AS-TuP5, 1

— L —

Lazik, C.: AS-TuP22, **4**
Leone, S.R.: AS-TuP14, 3
Lisowski, W.: AS-TuP23, 4
Liu, Y.: AS-TuP9, **2**
Luh, D.-A.: AS-TuP24, **4**

— M —

Macak, K: AS-TuP2, 1
Madan, A: AS-TuP11, 2
Meyer, III, H.M.: AS-TuP7, **2**
Moellmann, V.: AS-TuP8, **2**

— N —

Nakatani, K.: AS-TuP20, 4

— O —

Ohashi, Y.: AS-TuP20, 4

— P —

Page, S.C.: AS-TuP3, 1
Pinto, T.L.: AS-TuP11, 2
Piper, L.F.J.: AS-TuP18, 3

— R —

Raman, R.: AS-TuP9, 2
Reece, C.E.: AS-TuP6, 1
Rees, J.A.: AS-TuP12, 3
Ribeill, G.: AS-TuP6, 1
Roberts, A.J.: AS-TuP2, 1
Ronsheim, P.: AS-TuP11, 2

— S —

Sakai, Y.: AS-TuP19, **3**
Sanada, N.: AS-TuP20, 4
Sekiguchi, T.: AS-TuP5, 1
Seymour, D.L.: AS-TuP12, 3
Smith, K.E.: AS-TuP18, 3
Smithers, M.A.: AS-TuP23, 4
St. Lawrence, B.M.: AS-TuP11, 2
Streubel, P.: AS-TuP1, 1
Surman, D.J.: AS-TuP2, **1**
Suzuki, M.: AS-TuP20, 4

— T —

Tai, T.L.: AS-TuP11, 2
Takahashi, L.K.: AS-TuP14, **3**
Takaishi, R.: AS-TuP19, 3
Terry, L.: AS-TuP22, 4
Tian, H.: AS-TuP6, 1
Titz, T.: AS-TuP8, 2
Tougaard, S.: AS-TuP25, **5**
Tsuei, K.-D.: AS-TuP24, 4
Turansky, A.R.: AS-TuP11, 2

— U —

Uritsky, Y.: AS-TuP22, 4

— W —

Whitmore, T.D.: AS-TuP12, 3
Wierman, K.W.: AS-TuP21, **4**
Wilson, K.R.: AS-TuP14, 3

— Z —

Zhang, Y.: AS-TuP18, **3**
Zhou, J.: AS-TuP14, 3
Zhu, Z.: AS-TuP11, 2
Zuo, J.: AS-TuP8, 2